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X-RAY SPECTOGRAPHIC ANALYSIS — a new technique in wood preservation research

By D. S. BELFORD*, B.Sc., Ph.D.

A review of the principles, practice and application of this technique, based on a paper delivered at the B.W.P.A. annual convention at Cambridge earlier this summer.

DURING THE PAST two decades physicalt echniques have been applied on an increasing scale both to fundamental research and to routine industrial control. Although the basic theory underlying these techniques has, in many cases, been known for some years, it is only as a result of comparatively recent advances in electronics and instrumentation that such methods have resulted in commercially available equipment.

A considerable number of physical methods such as electron microscopy, X-ray diffraction analysis and electron diffraction analysis have already been used as powerful tools for research into the distribution of preservatives in wood, as discussed by Professor Preston at the 1959 B.W.P.A. Convention and elsewhere (Belford 1960, Preston 1959, Belford, Preston, Cook and Nevard 1959). It is hoped here to show how another physical technique, X-ray spectrographic analysis, can be applied to rapid routine analysis which is required both for research and routine control in the wood preservation industry. Analysis is needed for the determination of preservative distribution, permanence and loading, and because of the variability with which wood, in common with other natural products, is endowed the work on any research project is statistical in nature and calls for a large number of separate determinations to ensure significant results.

Standard chemical techniques are available for the estimation of chemicals used in wood preservation, but for the examination of preservative treated wood, lengthy acid-digestion or extraction procedures are normally required. The time taken for analysis together with the many manipulative operations involved, thus create a serious bottleneck in any large scale experimental programme, and for this reason some physical method was sought which should, if possible, have the following attributes: (1) no acid-digestion or extraction, (2) minimum of laboratory manipulation, (3) non-destructive, and (4) rapid.

Principles of X-ray Spectographic Analysis

Only X-ray spectrographic analysis was found fully to meet these requirements. The technique has been applied in recent years to many analytical problems in the fields of alloys, pure metals, cements, oils, ores, basic chemicals and plastics. The first application to wood preservation was by Wright & Storks (1957) for the estimation of pentachlorophenol in wood, and the conclusions of these workers will be considered later in the present paper.

In the X-ray spectrograph the sample, which may be solid, liquid or powder is irradiated with a high energy beam of X-rays, and each element present in the sample is excited to produce its characteristic X-rays. These consist of radiations whose wavelengths are unique to the individual elements present in the sample and are emitted simultaneously. When separated by means of an analysing crystal each wavelength is identifiable with a particular element, and its intensity indicates the relative amount of that element in the sample. A detailed consideration of the principles of this technique is beyond the scope of the present paper, and a full account is given elsewhere (Liebhafsky *et al* 1960, Birks 1959). The operation of the instrument is indicated in Fig. 1, and may conveniently be considered in three stages, namely, excitation of the characteristic X-rays, dispersion according to wavelength, and detection and measurement.

EXCITATION—Barkla and Sadler (1908) first demonstrated that secondary X-rays were emitted when an element was bombarded with a beam of primary X-rays. This characteristic X-radiation from an element present in the specimen is produced only when the energy of the beam of incident X-rays from the X-ray tube is sufficient

* *Hickson's Timber Impregnation Co. (G.B.) Ltd.*

to knock out the inner electrons of the atoms of that element. If one of the electrons is removed from one of these higher energy levels an electron from one of the lower levels must reoccupy the higher level to restore equilibrium. The energy of this transition is released as an X-ray quantum having a wavelength dependent upon the difference in energy of the electron in the initial and final states.

K-series lines are obtained when a K-electron is ejected and replaced by electron transitions from the outer shells to the K-shell.

The number of lines depends on the number of lower energy electron levels available for the reverse transitions, and increases with increasing atomic number.

Two factors make this characteristic or "secondary" radiation produced from the sample of particular interest to the analysis:

1. The relative simplicity of the characteristic X-ray spectra, which is due to the limited number of energy levels in the atom and to certain prohibited transitions.

2. The independence of the characteristic X-ray spectra of the molecular state or chemical form of the element undergoing excitation. This is because the X-ray lines come from the inner electrons of the atoms, the outer valency electrons playing no part.

In the X-ray spectrograph the characteristic X-ray spectra of the elements present in the sample are emitted simultaneously and before any systematic examination can be carried out it is necessary to distinguish the various wavelengths.

DISPERSION—This is normally accomplished by means of diffraction from a flat analysing crystal. W. L. Bragg (1913) demonstrated that such diffraction could be simply explained by the well known law bearing his name—

$$n\lambda \times 2d \sin\theta$$

where λ is the wavelength of the reflected radiation, θ is the angle of incidence, d is the interplanar spacing of the crystal and n is the order of the reflection. Reflection of X-rays will only occur when this relationship is satisfied, and so for a given crystal, X-rays of a certain wavelength will only be reflected at one angle, the value of which may be calculated.

In the normal flat crystal spectrograph, part of the beam of characteristic X-rays which is emitted from the sample in all directions, is limited to a parallel beam by a collimator consisting of an array of parallel metal plates. The collimated beam then strikes the analysing crystal which may be rotated about its axis as indicated in Figure 1. For each setting of the crystal only one wavelength will be reflected according to Bragg's law as outlined above.

DETECTION—Having found a means of separating the collimated beam of secondary X-rays from the sample into its constituent wavelengths, some way must be provided to detect and measure the intensity of the characteristic lines originating from the sample.

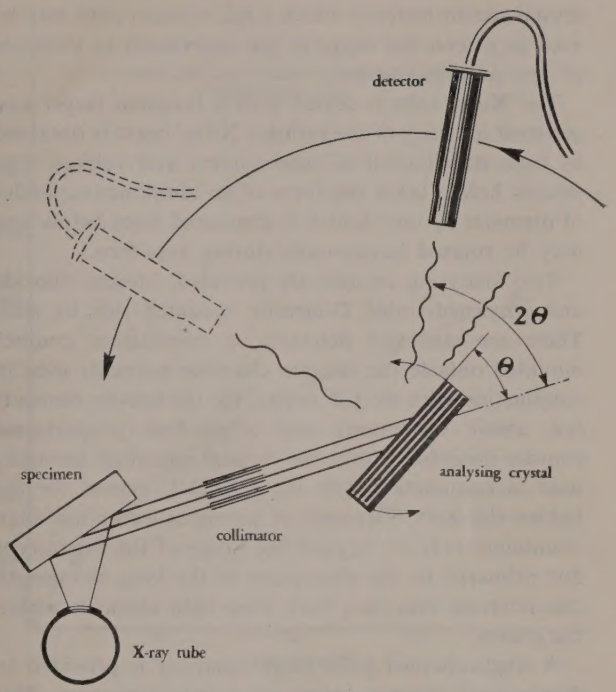

Fig. 1. Diagram showing the operation of X-ray spectrometer.

The recent widespread application of X-ray spectrographic analysis is primarily due to the development of counting tubes as detectors, and Geiger, proportional and scintillation counters have largely replaced photographic film for this purpose.

In the spectrometer the detector is arranged to rotate in the angular ratio of 2:1 as indicated in Fig. 1.

The line radiation is reflected from the crystal into the detector where the energy of the X-ray quanta are converted into electrical impulses or "counts". After amplification the impulses are fed into an electronic counting panel where they can be either integrated by a rate meter and charted on a graph recorder, or scaled down on to electronic counting tubes for visual reading.

Thus rapid qualitative analysis may be carried out by "scanning" the secondary X-rays emitted by the sample, continuously rotating the crystal and detector, and recording the characteristic lines on the graph recorder. Quantitative work may be carried out by setting accurately the crystal and detector to intercept a characteristic line of a given element and then measuring its intensity in terms of cts./sec., in comparison with chemically analysed standard samples.

Equipment

The work described in this paper has been carried out on the Philips All-Vacuum Spectrograph. This is a plain

crystal spectrometer in which a full vacuum path may be used to extend the range of the instrument to elements of low atomic number.

The X-ray tube is sealed with a tungsten target and constant intensity of the primary X-ray beam is obtained by high stabilisation of tube current and voltage. The sample holder takes the form of an aluminium cylinder of diameter 32 mm. which is irradiated from below and may be rotated horizontally during exposure.

Two analysing crystals are provided, lithium fluoride and ethylenediamine D-tartrate mounted side by side. There are also two detectors, a scintillation counter mounted outside the vacuum chamber normally used in conjunction with the LiF crystal for the heavier elements (i.e. above vanadium), and a gas-flow proportional counter mounted within the vacuum chamber, normally used in conjunction with the E.D.D.T. crystal for the lighter elements. Elements of atomic number less than aluminium (13) are beyond the range of the instrument due primarily to the absorption of the long wavelength characteristic radiation from these light elements within the system.

A single-channel pulse-height analyser is provided to discriminate against unwanted scatter radiation. This is particularly useful in the case of light element work.

The electronic recording panel has the normal chart recorder and counting facilities.

Qualitative Analysis

The technique is extremely useful for the simple qualitative examination of solids, liquids or powders. Samples of treated wood are merely cut to a convenient size and placed in the sample holder, while samples of powders or solutions are supported by a thin low-absorption Mylar polyester film window. In all cases the samples are irradiated from below. In order to determine which elements are present in the sample a chart recording is made. The angle (θ) between the surface of the analysing crystal (Fig. 1) and the beam of secondary X-rays from the collimator is progressively increased, and at given angles the corresponding characteristic lines of the elements in the sample are reflected.

The detector and crystal are rotated automatically in the ratio of 2:1 and variations in reflected intensity are integrated and plotted on the chart recorder as a series of peaks.

An example of such a recording from a sample of wood treated with a zinc-copper-chrome-arsenate formulation is shown in Fig. 2. A second pen on the recorder indicates the angle (2θ), so that a particular peak may be identified from the angle at which it appears. This identification is facilitated by tables (Powers 1957) which have been prepared showing values for 2θ corresponding to characteristic wavelengths using various analysing crystals.

In Fig. 2 it will be noted that the zinc, copper, chromium and arsenic K α lines may readily be identified. The remaining peaks are either lower order reflections from these elements or are the characteristic lines from the tungsten target of the X-ray tube. It will further be noted that these peaks occur above a background level of "counts" which is mainly due to scattering of the primary beam within the sample. The height of a peak of a given element is proportional to its concentration in the sample, but different elements cannot be compared in this way.

In the field of wood preservation and treatment, the following elements of common interests may readily be identified: tin, arsenic, zinc, copper, chromium, chlorine, sulphur and phosphorus, the latter two elements occurring primarily in flame retardant formulations. Two further elements which are sometimes used for wood preservation namely boron and fluorine are outside the

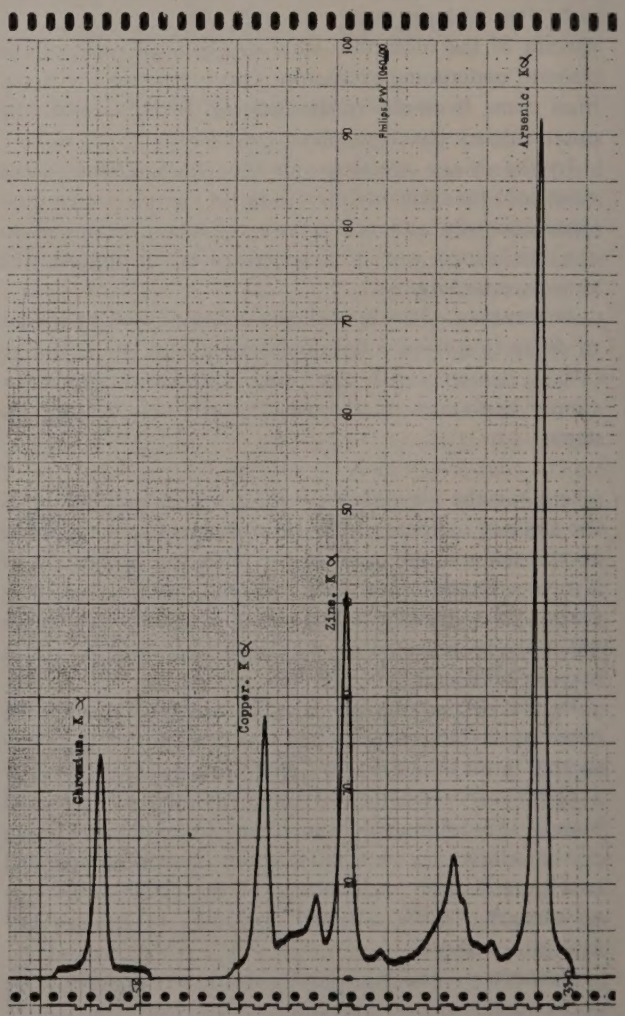

Fig .2. Chart recording made on a sample of wood treated with a zinc-copper-chrome-arsenate type preservative.

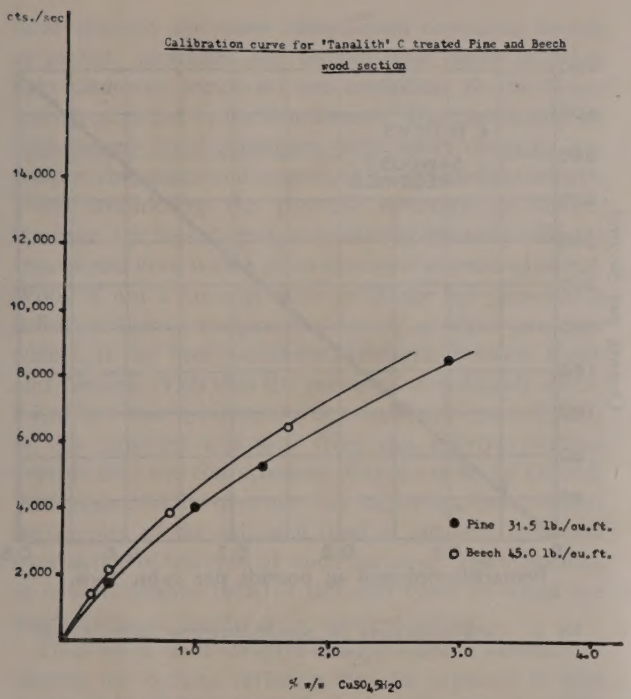

Fig. 3. Calibration curve for $CuSO_4 5H_2O$ in redwood and beech wood sections.

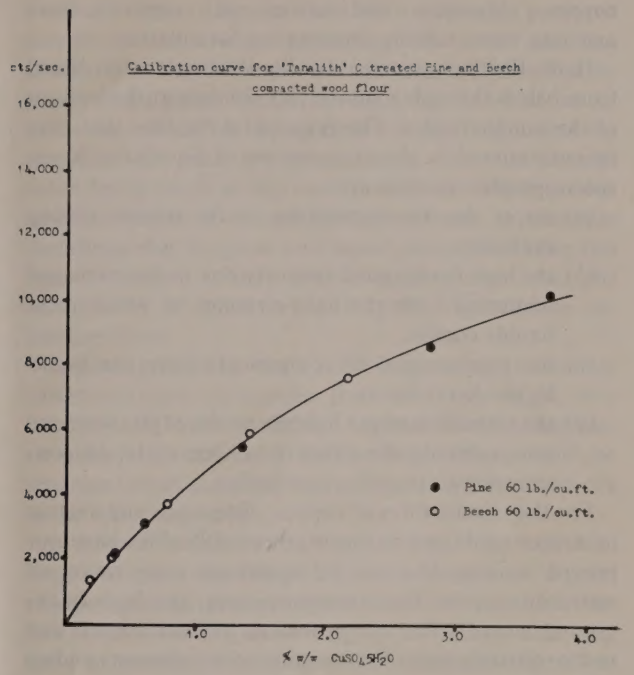

Fig. 4. Calibration curve for $CuSO_4 5H_2O$ in redwood and beech compacted flour wood pellets.

range of commercially available equipment as are many of the organic compounds consisting largely of carbon, hydrogen, oxygen and nitrogen e.g.—creosote, but in the case of the elements listed above, a few minutes automatic scanning on the instrument will generally suffice to identify the type of preservative.

Quantitative Analysis

For quantitative analysis the intensity of the characteristic radiation of a given element must be related to the per cent. composition of that element in the sample. The line intensity is determined either by collecting "counts" for a certain period of time (fixed time technique) or by measuring the time taken to collect a certain number of counts (fixed count technique). We have normally used the former method and it is usual to measure the intensity at the peak angle and at a suitable "background" position adjacent to the peak. The true intensity "peak minus background" is then related to the concentration of that element.

Although the relationship between intensity and composition is more nearly linear in X-ray spectrography than in other forms of spectroscopy (e.g. emission) direct quantitative analysis cannot be carried out. The controlling factor in all quantitative work is the "matrix" effect that varies with different types of sample and must be overcome by the use of calibration standards.

Absorption effects occur because characteristic X-rays are excited below as well as at the surface of the sample. Thus both the primary and the characteristic X-rays must pass through increasing amounts of the matrix material with increasing depth. The absorption coefficient for X-radiation (discounting absorption-edge effects) increases with atomic number and increasing density of the sample, thus changes in composition between samples will influence the depth of penetration of the primary beam and the depth from which secondary radiation can emerge.

A second form of absorption effect can occur in certain cases when the characteristic lines of some of the matrix elements excite the characteristic spectrum of the element under examination causing enhancement.

To carry out a quantitative analysis it is necessary to obtain a relationship between line intensity and concentration of an element. The simplest method, which is most usually adopted, is to prepare a calibration curve showing the variation in line intensity with concentration using the chemically analysed or gravimetrically prepared standards.

Experimental Procedure

ANALYSIS OF SOLUTIONS—One of the first applications of X-ray spectrographic analysis to liquids was the determination of lead and bromine in petrol (Birks *et al* 1950). The work described in the present paper has primarily been concerned with the determination of

copper, chromium and arsenic in copper-chrome-arsenate water soluble preservative formulations.

In the Philips instrument the liquid sample is irradiated from below through a thin Mylar window at the bottom of the sample holder. The principal difficulties that may be encountered in the examination of liquids by X-ray spectrographic analysis are:

(*a*) errors due to evaporation of the solvent during exposure.

(*b*) the high background intensity due to the increased scattering from the light elements of which most liquids consist.

(*c*) the precipitation of components from the liquid by the X-ray beam.

(*d*) the formation of gas bubbles on the Mylar window, thus reducing the effective volume of liquid contributing to secondary excitation.

For the examination of copper, chromium and arsenic in aqueous solution, however, these difficulties have not proved serious. There is no significant evaporation of water during the short exposure time, the high background intensity has not proved an embarrassment due to the relatively high concentration of the elements undergoing excitation and again, during the short exposure time no significant precipitation or occluded gas bubbles have been found to occur. It is important, however, to ensure that the solutions examined are clear and contain no suspended sediment, since if such a sediment containing the elements in question settles on the Mylar window during irradiation an abnormally high value would be obtained.

For analysis, 10 ml. of a gravimetrically prepared standard solution of the copper-chrome-arsenate preservative solution (normally in the range of 1-5 per cent. w/v total salt content) is pipetted into a clean sample holder. The sample holder is placed in the spectrometer and the peak minus background value determined for copper, chromium and arsenic in turn by accumulating counts for 64 seconds. This procedure is repeated on two further standard solutions covering the range of the unknown samples and a calibration curve prepared. Having established this curve, unknown samples may readily be analysed following the same procedure as outlined above, and from the peak minus background value for each element the per cent. w/v of that element may be obtained from the calibration curve. The instrument is normally calibrated twice a day and after calibration unknown samples may be analysed for copper, chromium and arsenic at the rate of one every eight minutes. This is a substantial saving in time over conventional methods of analysis.

The examination of phosphorus in flame retardant formulations is slightly more complicated since the estimation of phosphorus in common with other light elements must be carried out under total vacuum conditions, in view of the high absorption of the phosphorus K α radiation in air. It is impracticable to examine aqueous solutions direct due to excessive loss of water and splashing due to the out-gassing of air from the solution. In addition, the thin Mylar window used for solution work absorbs a substantial proportion of the phosphorus K α radiation under these conditions. For the analysis of phosphorus in aqueous solution, therefore, we have adapted a technique originally proposed by MacNevin (1954) and used filter paper which has been impregnated with the solution to be analysed and then dried. The dried sample of filter paper may then be examined in the spectrometer satisfactorily under total vacuum conditions.

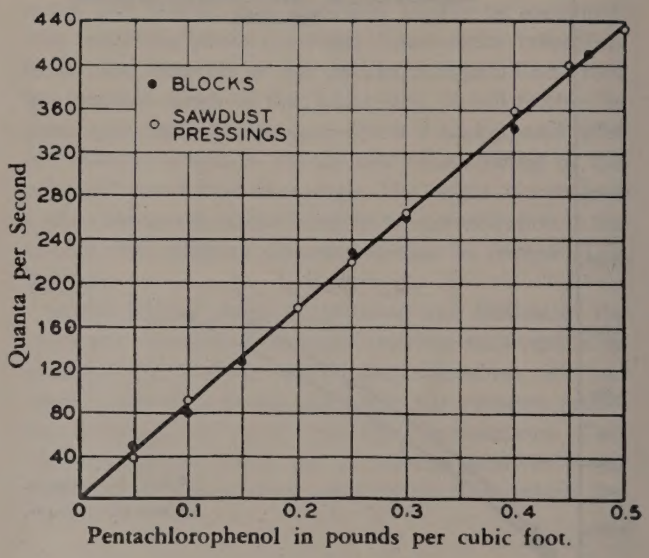

Fig. 5. Calibration curve for pentachlorophenol (after Wright Storks 1957).

The filter paper used for this purpose is Whatman's number 40, and a small standard amount of the solution under test is placed on a filter paper disc by means of a micro-pipette. The spread of solution on the filter paper is limited to the area of the X-ray beam by means of a wax ring of diameter 15 mm. which is imprinted on the filter paper disc by means of a hot cork borer (number 12) which has been dipped in molten wax. In this way calibration curves may be obtained as outlined above and a satisfactory degree of reproducibility is obtained. Although the time saving is not as spectacular as in the case of direct solution analysis, the technique is useful in the examination of a large number of samples.

ANALYSIS OF TREATED WOOD—Considering preservative treated wood as a matrix for X-ray spectrographic analysis both the chemical and physical composition must be considered. The chemical composition may be quickly disposed of since an elemental analysis, timbers

have virtually the same composition consisting largely of carbon, hydrogen and oxygen with traces of other light elements, which do not contribute to the X-ray spectra recorded by the instruments. Thus chemically we have largely ideal conditions with heavy elements *e.g.* copper, chromium and arsenic, in a light element matrix.

On considering the physical structure of timber, however, the high degree of variability between different species and even with a given species is at once apparent. Wood is not a homogeneous substance but possesses a definite cellular structure, the density of which can vary widely. It has been established (Belford, Preston, Cook and Nevard 1959) that the preservative is largely distributed as a micro-crystalline deposit within the cell walls of this structure and it is from this micro-crystalline deposit that the characteristic X-rays are to be excited. With regard to density, one very important factor is that the density of the cell wall itself is virtually the same irrespective of the type of wood involved. The variations in overall density between different types of wood are mainly due to variations in micro-structure.

Thus while chemically, wood provides a satisfactory matrix, the density variation may be expected to give rise to some absorption effects in X-ray spectrographic analysis. There is yet a further variation to be considered in addition to variable density. There is also a varying distribution of preservative within the sample due both to the overall gradient which results from vacuum/pressure treatment in common with other forms of treatment and to micro-variations due to wood structure. This micro-variation has been studied by means of a further X-ray technique, micro-radiography (Belford 1960).

To sum up the features of preservative treated wood in relation to X-ray spectrographic analysis, therefore:

1. The matrix is of a fairly uniform chemical composition largely made up of light elements *e.g.* carbon, hydrogen and oxygen.
2. The matrix is subject to density variations usually between 20-80 lbs./cu. ft. with a much smaller range for a given species.
3. This density is largely due to the relative size, thickness and shape of the wood cells since cell wall density remains reasonably constant in different timbers.
4. The preservative is located largely within these cell walls as a micro-crystalline deposit and the concentration can vary widely over a distance of a few millimetres.

In the development of our technique for handling this material two types of sample have been adopted, namely wood sections and compacted wood flour pellets.

Considering the wood sections first, these are of dimensions 0·5 in. × 0·5 in. × 0·15 in.; the length and breadth of the sample are not critical since it is placed over a 9 mm. diameter hole in a specially prepared sample holder always presenting the same area for irradiation. The thickness of each sample is checked by a go-not go gauge within the limits of 0·14-0·16 in. In order to obtain an integrated average value for the sample, both sides are irradiated in turn, the average value being taken as representative of the whole section and the sample is rotated during exposure to minimise variations due to grain and wood structure. Using this type of sample it may readily be confirmed that the distribution of preservative may be regarded as inhomogeneous.

In any application where fairly large sample to sample variations occur in samples from the same origin, it is important to make sufficient number of measurements so that statistical and instrumental errors may be separated from sampling errors. This may be carried out in the following way:

A strip of wood 0·15 in thick, 0·5 in. wide and 5 in. long was carefully selected from a sample of regular straight grained preservative treated timber which was free from knots and other irregularities. From this strip ten $\frac{1}{2}$ in. sections were prepared. Instrumental error was determined on the copper K $\propto$ line by replacing a given sample in the sample holder and re-setting the spectrometer between ten successive counts; the standard deviation for these ten measurements was then determined and agreed closely with the theoretical value calculated from the counting statistics.

A single count was then carried out on each of the ten samples and again the standard deviation was determined. In this case the observed standard deviation was of the order of ten times greater than that obtained from the repeated examination of a single sample. Thus sample to sample variation between adjacent sections, even in these ideal conditions, is far greater than any short term instrumental variations.

This type of sample is now used primarily to obtain detailed information on the relative proportions of preservative components so as to obtain data for salt gradient studies rather than to assess the overall loading of preservative in a given piece of wood, which would clearly be impracticable without excessive replication.

To summarise the results obtained on these wood section samples a calibration curve prepared for redwood at 30 lb./cu. ft. and beech at 45 lb./cu. ft. is shown in Fig. 3. It will be seen that in the case of copper K $\propto$ radiation, the results are slightly higher in the heavier sample.

The average loading of preservative

Leaving wood samples for the moment, the problem associated with determining the average loading of preservative in a large sample will now be considered. In conventional wet chemical analysis this situation is

dealt with by acid-digestion or extraction of a complete cross section of the sample. It clearly would be too time-consuming to carry out replicate analysis on a large number of small wood samples in order to arrive at the average loading of wood preservative and so for this purpose wood flour is used. A complete cross-section of the sample is split into small pieces and passed through a grinding mill reducing the sample to small particles. This grinding largely destroys the wood structure and the wood flour thus obtained is then compacted into pellets of uniform density, thus eliminating density effects.

0·25 gms. of wood flour is placed in a small mould of diameter of 1 cm. and a pressure of about 10 tons is applied while the sample is evacuated. After varying the conditions, it was found that uniform compacted discs at a density of 60 lb./cu. ft. could be prepared from both beech and redwood flour. A calibration curve for compacted wood flour pellets is shown in Fig. 4. By destroying the original wood structure and re-compacting a constant weight into a given volume, density effects may be discounted since as mentioned earlier the density of the wood substance remains virtually constant. For the determination of the overall loading of preservative these compacted wood flour pellets are normally used and the results obtained in terms of per cent. w/w of the element in question. This is then converted into conventional units of w/v (e.g. lb./cu. ft.) after determining the density of the original wood sample.

It is on this point that our results differ slightly from those obtained by Wright & Storks (1957). These workers have published a calibration curve shown in Fig. 5 for the estimation of pentachlorophenol in timber. Two points are at once apparent: first the units are expressed directly in terms of per cent. w/v (lb.cu. ft.) and secondly the intensity of the characteristic line appears to be independent of the method of sample preparation comparing wood sections with sawdust pressings. The authors state that the X-ray beam selects a standard volume thus eliminating tedious volume measurements. In our experience this statement can only be true for wood samples at a given density and the agreement between sawdust pressings and wood blocks may have been fortuitous in that they must have had a similar density or a difference in thickness balanced the density difference. The latter point explains the similarity between Fig. 3 and Fig. 4.

For these reasons we prefer to express analytical results obtained by X-ray spectrographic analysis in terms of per cent. w/w and subsequently to convert this to lb./cu. ft. after carrying out a density determination on the sample.

Turning now to consider the estimation of phosphorus in flame retardant treated timber, for this purpose compacted wood flour samples are normally used since the relatively long wavelength of the phosphorus K α line at 6·15 A is extremely sensitive to variations in sample density and surface finish. As was mentioned earlier, for phosphorus determination the full vacuum path of the instrument must be utilised with the gas flow counter and the E.D.D.T. analysing crystal. A useful increase in peak to background ratio is obtained by the use of the single channel pulse height analyser.

Applications

GENERAL—It will have become apparent by now that X-ray spectrographic analysis in the wood preservation industry will find the greatest application in the field of multi-salt water soluble preservatives or fire retardants. Many other compounds may be dealt with, however, *e.g.* pentachlorophenol, and qualitative analysis may be carried out on surface coatings.

The facility for rapid analysis is of such general value that it is difficult to single out any one application for special mention but two of particular interest have been selected for further discussion. The first concerns routine production control and the second an extensive research project that has recently been completed in our company's laboratories.

ROUTINE PLANT CONTROL—With the possibility of rapid solution analysis for copper, chromium and arsenic in aqueous solution, it becomes feasible to introduce a system of regular short term check analyses of our commercial plant solutions. The wood preservation industry is largely made up of a large number of small productive units in contrast to the centralised production facilities of many of the competitive materials with which timber is faced. Problems of tight production control are greater in the case of a large number of small units and X-ray spectrographic analysis has been used partially to solve this problem.

Small samples (25 ml.) are sent from the treatment plant by post to the central laboratory for checking on a bi-weekly basis in order that a constant check can be kept on the condition of all solutions in current commercial use.

The solution strength is normally estimated at the treatment plant by means of a hydrometer. This method while capable of accurate results when the correct procedure is followed, is liable to operator-error introduced by faulty technique, and gives no information on the general condition of the solution. X-ray spectrographic analysis examination rapidly determines the true solution strength, the balance of components and at the same time the pH of the solution may readily be checked. Thus, the solution used for treatment in plants situated in remote areas and different parts of the country may be subjected to the same rigorous quality control to that

which they would receive under conditions of centralised production. This service is now being extended overseas.

LARGE SCALE SAMPLING PROJECT—The X-ray spectrometer is also a powerful tool in wood preservation research. The speed of analysis which is available enables sampling projects to be undertaken which would have been impossible before its introduction due to the tremendous task of analysing large numbers of samples by traditional chemical techniques. Such a project, which may be used as an example of this, has recently been completed.

Wood which has been installed as packing in water cooling towers for power stations is required for economic reasons to last for 25 years or more (Ross and Wood, 1957). Basidiomycete attack does not occur under saturated conditions, but untreated wood is subject to a type of degrade known as soft rot which is of importance because of the thin sections employed. Although the average life is longer, some untreated packings have required replacement after only seven years. In view of the continuous washing to which the timber is subjected, it is clearly important to have a highly fixed form of preservative in order to give adequate protection against soft rot degrade.

New specification

After a considerable amount of laboratory work and field service trials a treatment specification (TC/3001/1) for cooling tower packing timbers was prepared by the British Central Electricity Generating Board. This specification covers the treatment of European redwood laths to an average retention of 1·25 lb./cu. ft. with either copper-chrome or copper-chrome-arsenate water-borne preservatives. While accelerated laboratory tests indicate that treatments carried out to this specification will result in a satisfactory degree of fixation giving the required service life, confirmation from commercially treated timber under actual service conditions would clearly be desirable, particularly with regard to the actual loss of salts after several years of operation.

In order to obtain detailed information on this point a comprehensive study has recently been carried out under the terms of a research contract awarded by the C.E.G.B. to investigate this problem. The C.E.G.B. co-ordinated the project and with the co-operation of the Atomic Energy Authority and the South of Scotland Electricity Board together with the manufacturers of the two preservatives concerned, twenty cooling towers were selected of varying age. One hundred slats were taken from the top layer of the packing from each tower at random and the pieces placed in a sealed bag and opened under supervision at the test laboratory. Because of the variable penetration of preservative into the heartwood, only sapwood slats were selected for analysis. After selection of the sapwood, ten samples were chosen using a list of random numbers taken from a sealed envelope. Two pieces one from each end of the sample were taken for analysis, and the wood section type of sample was used with the purpose of obtaining information on the salt gradient.

At the time of writing the data obtained in this project is still undergoing statistical analysis since over 12,000 separate determinations have to be assessed and correlated with operating conditions. A small number of determinations by X-ray spectrometer were checked independently by means of chemical analysis and a preliminary statistical check shows that there is no significant bias between the results obtained by X-ray spectrographic analysis and chemical analysis. A preliminary examination on the overall figures obtained has been made by taking an average of the results for each tower and expressing this average against the hours of operation.

For towers containing timber treated to the new specification, operating hours vary from 5,000 to 36,000 and the conclusions from this examination are clear, indicating that no significant loss of salts occurs in the treated timber even after continuous operation for 36,000 hours. As was expected there is evidence of slight loss in the first 5,000 hours operation but thereafter there would appear to be no further loss. The final conclusion from this work, however, must await the results of the more detailed statistical investigation that is to come.

Projects of this nature are of considerable value in confirming the results of accelerated laboratory tests, and further studies are contemplated.

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Near Nairobi, a team is identifying and classifying 120,000 eelworms in a drive to help local farmers overcome the problem of this pest which reduces some crops by as much as 25 per cent.

STUDYING NEMATODES IN EAST AFRICA

AT a microscope in room number seven in the modern laboratories of the East African Agriculture and Forestry Research Organization at Muguga, near Nairobi, a young scientist is working on a two-years research project, examining worms no bigger than pin-heads which are reducing the yield of African-grown crops by nearly one-quarter.

The scientist is Mr. A. G. Whitehead, a nematologist, and he is checking the first of 120,000 root-knot nematodes—eelworms—collected from fourteen areas of Kenya, Tanganyika and Uganda by his two Kikuyu laboratory assistants, classifying every one—twenty-five of the tiny worms laid side by side would cover no more than one inch—to prepare a simple and inexpensive plan for farmers to rotate their crops to defeat the ravages of the nematodes.

The eel-worms, slender, thread-like worms, exist in hundreds of species in East Africa, and Mr. Whitehead's research work includes the job of identifying them.

They have no eyes and are attracted to the plant roots by root secretions. They can be carried from place to place when soil is moved, or on farm implements or on people's feet. Once settled on the root they like, they start to draw its nourishing juices down to the roots, using a sort of hypodermic-syringe-like proboscis.

Eighty to ninety per cent of East Africa's crops are attacked by the eel-worms, and there may be between 50,000 and 100,000 million of them in an acre of soil. If they cannot find some staple crop, they will exist by sucking roots of wild plants. Result of their greed is shown by the bulging, knotty roots of the crops they live on, and they can bring the growth of the roots of plants to an abrupt halt. They live for about twelve months, producing eggs the whole time, but their life-cycles are still being studied at Muguga and elsewhere.

Mr. Whitehead pre-selected his local surveys, each of fifty square miles, where Africans were cultivating the land intensively, then selected within them ten sub-areas. In each sub-area a range of crops—tomatoes, maize, dwarf peas, pepper, tobacco, European potatoes, sweet potatoes, sugar-cane, cabbage, pumpkins and bananas—and common weeds are sampled by Mr. Livingstone Kariuki and Mr. Njenga Kinyanjui. Plastic bags and labels in their pockets, they travel the areas accompanied by an officer of the territorial Agricultural Department who explains to the local people the object of sampling, digging up the crops and the weeds.

His assistants aim at taking their root samples just before the crops are harvested in each district, at the time when, if they are attractive to the eel-worms, they are most likely to be infested.

Ten days digging

From each sub-area they take two samples of each crop and each weed, 400 samples from each area. Each root system taken to the laboratories at Muguga provides twenty specimens of eel-worms, so that from each 50 square miles area there are 8,000 nematodes.

After ten days of digging and labelling, the two Kikuyu finish their task, return to Muguga, wash and stain the roots, and put them in sealed glass tubes to await dissection, then set off again.

Their field work ended in July and they now have before them eighteen months laboratory work, painstaking extraction of the eel-worms from the knotty roots and cutting tail sections from each worm.

Twenty of these tail-end sections, which include the egg-pores of the worms, are carefully mounted on a glass slide ready for Mr. Whitehead to examine under his microscope.

Mr. Kariuki and Mr. Kinyanjui have returned to their laboratory to work on the dissections, doing a hundred each a day, seeking out the tiny worms by first

finding their egg-masses (500 to 3,000 eggs in each) then picking out the females and mounting a section of the eel-worms on slides.

Each 50-square-miles area sampled must be followed by forty days of laboratory work; Mr. Whitehead, identifying the species lined up on the glass slides by their finger-print like patterns, will take three weeks to work through the samples, averaging six hours every day, and doing up to twenty-five slides in each day's work.

He can identify the tail-end of the pear-shaped female eel-worm in from thirty to forty-five seconds if it is one of the main species. As he finishes his scrutiny he enters details in his files on each district, and so that agricultural officers and others can know quickly the results of the samplings, he writes up his findings for the agriculture and forestry journal published each quarter in East Africa.

This root-knot nematode survey of East Africa will prove whether or not the same range of species of the worms occurs throughout the three East African terri tories, or whether in areas with different climate and soils there may be a different range of species. Mr. Whitehead believes he will find a fairly set pattern, and will then be able to recommend growing of suitable nematode-resistant crops in East Africa as a whole.

Some species of eel-worms can only be found in certain crops, and above certain altitudes, And already, from the survey carried out not far from Muguga, in the Kikuyu area, it has been established that there are five or six main species of the crop-wasters, and that two of these species are common in the whole 50-square-mile area.

It is thought quite likely that there will be the same eel-worm picture all over East Africa. By the end of the survey the team will have enough positive information to be able to suggest selection of certain crops for certain localities and to prepare a map showing the eel-worm distribution.

It is known that some eel-worms prefer certain crops, that tomatoes, beans and pumpkins, for instance, are heavily attacked by the minute worms, but that they do not like maize. So, a farmer might be able to plant maize, which would attract no eel-worms and have clean ground for his next crop, so long as he kept it weed-free, for eel-worms like weed-roots. Then he could plant sweet potatoes or bananas in the clean ground, and, before the eel-worms, which are able to move through the ground very slowly, caught up with them, rotate his crops with maize again.

African farmers do not generally realise the damage caused by these eel-worms and the extent to which they reduce the yield of their crops—20—25 per cent. is not uncommon. Crops like beans and tobacco are usually more severely affected.

ORGANIC PHOSPHOROUS INSECTICIDES FOR THE CONTROL OF LIVESTOCK PESTS

By E. C. TURNER, Jr.*

One of the most significant trends in the control of livestock pests is the development of organic phosphorous insecticides applied directly to animals. This article—the first of two on the subject—discusses those which are safe for warm-blooded animals, and some which have been seen to exhibit systemic activity.

PRIOR TO 1942, livestock farmers relied mainly on inorganic chemicals and several natural organic insecticides for control of livestock pests. These chemicals were effective only in special circumstances and were not used to any great extent. Thus, control of these pests was generally limited to certain cultural and biological methods. The discovery of the insecticidal effectiveness of DDT and the related chlorinated hydrocarbons greatly expanded the use of insecticides for control of livestock pests. Farmers and stockmen at last possessed insecticides that could be applied both effectively and economically.

With the increased use of the chlorinated hydrocarbon insecticides, two rather disturbing factors began to appear. It soon became apparent that some insect pests were developing resistance to these compounds, an early example being the discovery of DDT resistance in the, common house fly in Sweden in 1947. Since that time this insect has become resistant to DDT in practically every country in the world. Pests of livestock such as ticks, blowflies and lice were also discovered to be resistant to one or more of the chlorinated hydrocarbon compounds in some areas. A review of the current status of insecticide resistance on livestock pests has recently been published by W. C. McDuffie[1].

The second disturbing factor appearing with the use of the chlorinated materials came with the discovery that these insecticides were stored in the milk, meat and fat of treated animals[2]. In the United States the presence of these residues in the tissues of animals has been regulated.

Preliminary investigations by toxicologists[3] have indicated that heavy dosages of parathion fed to dairy cows in the hay and orally in capsules was not found to be excreted in the milk, blood or urine. These factors and other investigations led research entomologists to investigate the use of organic phosphorous insecticides on livestock for control of parasites.

In reviewing the effectiveness of organic phosphorous insecticides against livestock, the writer is omitting their use against house flies and mosquitoes. This has been reviewed previously by A. W. Lindquist[4].

One of the characteristics of the early developed organic phosphorous insecticides was their extreme toxicity to warm blooded animals, particularly the compounds HETP (hexaethyl tetraphosphate) and TEPP (tetraethyl pyrophosphate). Laboratory tests and also accidental application of these compounds to livestock showed that the chemicals were extremely poisonous to warm blooded animals[5,6].

Included in this group of very toxic insecticides was parathion (*O*, *O*-diethyl *O*-*p*-nitrophenyl phosphorioate). This insecticide, though widely used for the control of plant feeding insects, was considered to be a hazard to workers handling the treated plants[7]. Careless use of this

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material resulted in human death on several occasions, and this high mammalian toxicity of the early developed organic phosphorous insecticides illustrates to some degree why entomologists were somewhat reluctant to use this group of chemicals for control of livestock pests.

Safe for animal application

Through the continued development of new organic phosphorous compounds for use against insects of all kinds, it was soon discovered that many of the new chemicals of this group were quite safe when applied to warm blooded animals. Actually some of these compounds were found to be safer than the highly regarded chlorinated hydrocarbon insecticides. Extensive studies by Dr. R. D. Radeleff were made at Kerrville, Texas, evaluating hundreds of chemical compounds on all types of animals. He summarized[8] the acute toxicity of chlorinated hydrocarbon and organic phosphorous insecticides and found that external application of most of the organic phosphorous compounds on one to two-week old calves resulted in toxicity equal to that of most of the chlorinated hydrocarbons. Other workers[9] reported that dogs fed 500 to 3500 mgs. per kgs. of malathion [O, O-dimethyl S—(1,2-dicarb=ethoxyethyl) dithiophosphate] showed no ill effects. In addition, the dipping of dogs in 2 per cent malathion resulted in no toxic symptoms, although it is well known that cats and birds are quite susceptible to many of our commonly used insecticides. However, Cross and Folger[10] demonstrated that malathion could be applied safely to cats in dosages large enough to kill fleas. Also, malathion could be placed in the cages of birds with no ill effects. These and many other examples demonstrated the safety of certain of the organic phosphorous materials, particularly malathion. Thus, the discovery of safe compounds led workers to begin tests on many ectoparasites of livestock. This work was expedited particularly on pests that were becoming resistant to the chlorinated hydrocarbon insecticides.

PARATHION—One of the first insecticides evaluated against pests of livestock was the compound parathion. It was characterized by high insecticidal activity against a wide range of insect pests at a very low dosage rate. Unfortunately, it also was characterized by rather high toxic activity against warm blooded animals. In spite of this rather undesirable characteristic, numerous investigators began testing this chemical against certain pests of livestock. Laboratory tests[11] against the sheep ked or tick, *Melophagus ovinus* (L.), demonstrated that parathion was highly effective at relatively low dosages. It was also reported[12] that a solution containing 1% of the insecticide applied to cages containing stable flies, *Stomoxys calcitrans* (L.) resulted in complete kill up to 14 days after exposure. The treatment, however, failed to kill the stable flies 50 days after treatment.

Parathion ground treatment for control of lone star ticks, *Amblyomma americanum* (L.), applied at the rate of 0.25 to 1 pound per acre gave control at least equal to chlorinated hydrocarbon insecticides applied at the rate of 1 to 4 pounds per acre[13].

In evaluating the toxicity of parathion to screw worm larvae, *Callitroga hominivorax* (Carl.) *in vitro*, Eddy[14] reported that 100 per cent mortality of newly hatched larvae was obtained with parathion dilutions of 0.00005 to 0.001 per cent. Against full grown larvae, 100 per cent mortality was obtained with solutions of 0.05 per cent to 0.1 per cent Dutoit & Fielder[15] reported that the exposure of *Lucilia cuprina* (Weid.) larvae to 6 parts per million parathion on filter paper resulted in 100 per cent mortality.

Using a spot treatment method in which only a small area of each cow was treated, Smith and Richards[16], found that 0.05 and 0.01 per cent concentrations of parathion remained effective against the short nose cattle louse, *Haematopinus eurysternum* (Litz.) for three weeks.

It will be noted that much of the early work with parathion was done "in vitro". These examples serve to emphasize that fact that while entomologists were quite impressed by the insecticidal effectiveness of parathion they were still somewhat reluctant to treat warm blooded animals with this chemical. It remained for other organic phosphorous insecticides to be evaluated against livestock ectoparasites.

Highly effective but less toxic

MALATHION—In an effort to find an insecticide of the wide insecticidal effectiveness of parathion but without its high mammalian toxicity, the compound malathion was developed and evaluated. Malathion was found to be satisfactory for most of the requirements for use on livestock. The compound was highly effective against a wide variety of insect pests and at the same time it was less toxic to warm blooded animals than most of the chlorinated hydrocarbon insecticides. Today, this chemical is probably one of the most important insecticides used in the control of livestock pests.

Malathion is highly effective against most species of lice attacking livestock. Tests by Smith and Richards[16] evaluating several organic phosphorous compounds have shown that malathion was effective not only against lice of cattle but lice on poultry and goats. DeFoliart[17] obtained excellent control of four species of cattle lice using a 0.5 per cent spray of malathion on cattle. Similar results against the long nosed cattle louse, *Linognathus vituli* (L.), was obtained by Anthony[18].

Against hog lice, *Haematopinus suis* (L.) Johnson[19] found that a 1 per cent spray resulted in complete control for more than 30 days after treatment.

Field tests by other workers[20] evaluating several organic phosphorous compounds for control of three species of goat lice also demonstrated excellent control with malathion.

Skerman[21] reported that this insecticide used as a dip in a 0.01 per cent solution resulted in at least 16 weeks control of the sheep body louse, *Damalinia ovis* (L.).

The use of malathion for the control of sheep keds, *Melophagus ovinus* (L), has been reported many times[22,23]. Malathion applied either as a spray or as a dip was quite effective against this pest of sheep.

Malathion along with several other phosphorous compounds has been evaluated for control of ticks on cattle in various countries. Legg[24] reported that a 0.5 per cent spray solution resulted in excellent control of *Boophilus microplus* Canestrini. Drummond *et al.*[25] obtained excellent control of the winter tick, *Dermacenter albipictus* (Pack.) using a 0.5 per cent spray solution on cattle.

Of considerable interest to entomologists was the effectiveness of organic phosphorous compounds against blow flies causing sheep strike. Workers in Australia and South Africa found that while malathion treatment of sheep gave good control of the blow fly larvae, the material did not last as long as some other organic phosphorous insecticides[26,27].

Malathion as an acaricide for control of mange mites has been used successfully in certain cases. Branden[28] reported that a 0.2 per cent dip solution resulted in successful control of sheep scab. Against scab mites on hogs, *Sarcoptes scabiei suis* (Gerlach), Raun and Ahrens[29] reported complete control in 19 days after treatment with 0.5 per cent or 1 per cent malathion spray.

Unpublished data by the writer indicated that 4 per cent malathion dust applied to the backs of dairy cattle resulted in control of horn flies *Siphona iritans* (L.) for 2 to 3 weeks. This has been confirmed by numerous workers in the United States.

Use against poultry pests

Probably the most widespread and successful use of malathion has been in the control of ectoparasites of poultry. Not only has this material been effective against a wide "spectrum" of poultry pests, but it has been found to be extremely safe to use on various domestic birds. Entomologists have reported this material to be effective against most mites and biting lice of chickens, turkeys, game birds and pigeons[30,37]. So effective has been this material that it has generally replaced the chlorinated hydrocarbon insecticides for control of poultry pests.

DIAZINON: [*O*, *O*-diethyl *O*-(2-isopropyl-6-methy-4-pyrimidyl) thiophosphate]—As an insecticide, diazinon is much more toxic to warm blooded animals than malathion. It is characterized by high insecticidal activity against certain insects over a rather long period of time and at a fairly low concentration. In fact the insecticide, though different from the chlorinated hydrocarbon insecticides, possesses comparable insecticidal activity to such compounds as aldrin and dieldrin. Riches and O'Sullivan[27] reported that diazinon dips resulted in longer effectiveness at lower dosages than did either aldrin or dieldrin against sheep blow flies. Branden[28] reported excellent control of sheep keds and lice with dilutions of 0.0005 per cent diazinon applied as a dip. He also stated that diazinon might prove superior in effectiveness against blow fly strike than either dieldrin or aldrin.

Skerman[21] in evaluating several insecticides for control of the body lice of sheep, *Damalinia ovis* (L.) reported that diazinon resulted in long lasting control at extremely low dosages. Again diazinon proved to be slightly superior to any other insecticide. This was substantiated by workers in Australia and South Africa[26,38,39].

Legg[24], in experiments evaluating diazinon against DDT and toxaphene resistant cattle ticks as well as normal ticks, found that this insecticide applied as a spray in dilutions as low as 0.05 per cent killed all ticks on the treated animals. The residue on the animal, however, only lasted for 1 to 2 days. No difference in toxicity was found between the DDT or toxaphene resistant ticks and the normal ones.

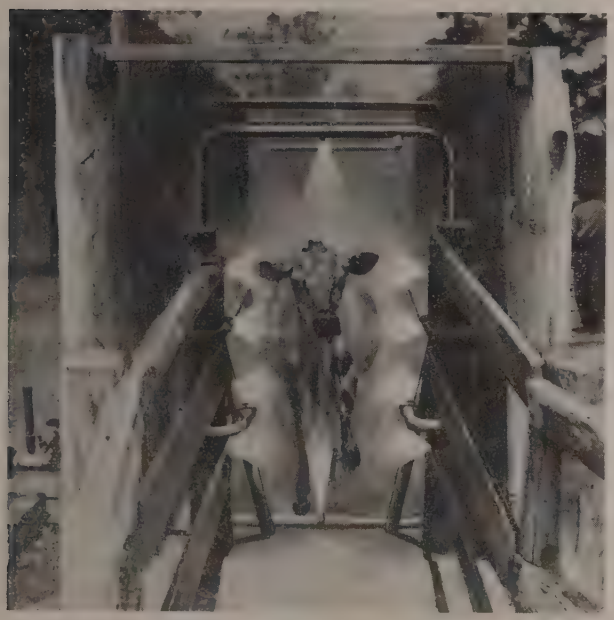
Effective for ten days after use

The effectiveness of diazinon against house flies in dairy barns is well known. Wingo[40] reported that good kill of stable flies, *Stomoxys calcitrans* (L.), was obtained for at least 10 days after dairy barns were treated for house fly control.

This outstanding success against insect pests especially at extremely dilute dosages would indicate that this material would certainly be one of the most widely used insecticides on livestock. Such is not the case. Here again entomologists are somewhat reluctant to use diazinon on warm blooded animals. It is the opinion of the writer that much more work needs to be done in the evaluation of this material against livestock.

OTHER ORGANIC PHOSPHOROUS COMPOUNDS—Several important organic phosphorous insecticides have been developed recently that show promise in the control of livestock pests. Among those in general are: *O*, *O*-diethyl-*O*-3-chloro-4-methyl-2-oxo-2*H*-1-benzopyran-7-yl phosphorothioate, also referred to in the literature as Bayer 21/199, coumaphos Co-Ral, Muscotox and Asuntol; dimethyl (2,2,2-trichloro-1-hydroyethyl) phosphate, also known as Bayer L13/59, trichlorphon Dipterex, Dylox, and Neguvon; and O,O-dimethyl-0-2,4,5, tri- chlorophenyl phosphorothioate, which may be called, fenchlorphos, ronnel, Dow ET-57, Dow ET-14, Korlan, Nankor, Trolene, Etrolene and Viosene. The synonomy of names

Below: A great deal of skill, experience and labour is required to dip sheep and cattle but modern apparatus such as the Cooper-Allman Spray Race, seen in use in the photograph, is easier to handle and more economical.



Above: Sheep scab in an advanced stage. This dreadful disease is caused by the mite Psoroptes communis. It has been virtually eradicated from sheep in Britain, Australia and some other parts of the world by the use of acaricides.

of these compounds can be obviously confusing to one not familiar with the literature, therefore, for the sake of simplicity in this article, the names Bayer L13/59, Bayer 21/199 and ronnel have been arbitrarily selected as representing the compounds.

The above insecticides are somewhat unusual in that they have been discovered to be effective systemically as well as being excellent external insecticides. Their importance in the field of animal systemics is increasing and will be discussed later.

Smith and Richards[16] evaluating organic phosphate insecticides against insect pests of cattle and goats obtained promising results. Against two species of goat lice, *Bovicola caprae* (Gurt.) and *B. limbatus* (Gerv.), Bayer L13/59 as a 0.01 per cent dip and Bayer 21/199 as a 0.002 per cent dip showed excellent residual effectiveness. In addition, these materials applied in spot treatments for control of cattle lice were highly effective.

The above insecticides have only recently been evaluated with regard the control of ticks. Research entomologists reported that these materials were quite effective for a short period of time. A 0.25 per cent spray of Bayer 21/199 and ronnel, when used against the winter tick, *Dermacenter albipictus* (Pack.), resulted in 100 per cent control for one week after treatment. The number of adults remained low at subsequent examinations for at least three weeks thereafter[25]. Seifert[41] evaluating Bayer L13/59 against ticks in Peru found that the material was quite effective initially but did not last very long. In laboratory and field trials in various parts of South Africa for control of cattle ticks including the arsenic, BHC, toxaphene, and DDT resistant blue tick, *Boophilus decoloratus* Koch, Fielder[42] demonstrated that Bayer 21/199 was a very suitable agent for the control of all cattle ticks. This material was applied weekly as a dip at concentrations of 200 parts per million. In field tests with a number of insecticides for control of lone star ticks, *Amblyomma americanum* (L.). Drummond and others[43] concluded that a 0.5 per cent spray of Bayer 21/199 would result in satisfactory reduction for 1 week after treatment. Other insecticides such as ronnel, as a 0.75 per cent spray and malathion, as a 0.5 per cent spray, were slightly less effective. Bayer L13/59 treatment resulted in unsatisfactory control.

Control of the ticks, *Rhinpicephalus bursa* Can-Fan. and *R. evertisi*, Neum. with Bayer 21/199 in dips of 100 to 225 parts per million was reported by Behrenz[44]. He also obtained control of *Amblyomma cajennense* Fab. and

A. hebraeum Koch., at a dip concentration of 450 parts per million.

Fields trials for the control of sheep keds using several phosphorous compounds have been conducted by Roth and Bigley[22] and also by Pfadt[23]. Sprays containing either 0.1 per cent Bayer 21/199 or 0.1 per cent ronnel resulted in complete control of the keds for at least 6 weeks after treatment. Hand dust applications of 5 per cent ronnel resulted in complete eradication.

Although the chlorinated hydrocarbon insecticides such as DDT, toxaphene, methoxychlor, and others are still highly effective against horn flies, some field tests have been carried out to determine the effectiveness of new organic phosphorous insecticides against this insect. Brundret *et, al.*[45] tested Bayer 21/199 as 0.25 and 0.5 per cent sprays. They found that the chemical afforded protection for about 3 weeks or about the same as a 0.5 per cent methoxychlor spray. Roberts[46] confirmed this in reporting that ronnel and Bayer 21/199 applied as a 0.5 per cent spray was as effective as methoxychlor and in some cases slightly better. Unpublished field tests by the writer also substantiated these results.

These new insecticides have also been evaluated against poultry pests with very favourable results. Linkfield and Reid[47] found that 5 per cent dusts of ronnel brought about complete eradication of the northern fowl mite, *Bdellonyssus sylviarum* (C & F). They also reported that the material was effective in the eradication of four species of biting lice. Kraemer[48] reported on field tests using several of the recently developed insecticides. Ronnel applied as a 0.5 per cent spray was promising but inconclusive against fowl tick, *Argus persicus* (Oken). Hoffman[49], in screening tests against three species of lice and the northern fowl mite, found that Bayer 21/199 achieved results comparable to malathion. Knapp and Krause[50] applied dust formulations of Bayer 21/199, Bayer L13/59, and ronnel to laying hens with effective control of the northern fowl mite for up to 28 days after treatment. They concluded that any one of these materials would result in complete eradication if all birds in a flock were treated.

Several other compounds are currently being evaluated for external application against livestock pests, but they have not come into general use as yet. The final marketing of new insecticides is often delayed due to the high cost involved in the initial research and developement. These new compounds may or may not compare well with existing materials.

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Next month: METHODS OF APPLICATION

Studying knockdown activity of pyrethrum

A STUDY is being made by the Tropical Products Institute in London of the evaluation of the quick "knock-down" characteristic of the insecticidal activity of pyrethrum, for the four esters alone and in the presence of a synergist. This is one of the lines of work being pursued by the institute in the field of insecticides, and revealed in its annual report for 1960 which has just been published.

The lines investigated were the knock-down and recovery of houseflies treated with pyrethrum extract, alone and when synergised with piperonyl butoxide, and the knock-down and toxicity curves of the pyrethrins and cinerins alone and when energised with piperonyl butoxide.

Effect of time

From the results obtained (says the report) it was possible to determine the effect of the passage of time on flies treated with a known quantity of pyrethrum, and the dose of active material needed to affect a given percentage of insects (usually 50 per cent) at a given time. The latter figures were plotted against time to give 50 per cent toxicity curves.

In general, at a fixed date, the toxicity curves of pyrethrum and its constituents are composed of four portions:

1. A very short latent period, during which a few insects only are knocked down.
2. The knock-down period during which the number of insects affected rapidly increases to a maximum.
3. The period of maximum effect.
4. The period of recovery.

The duration of each period depends on the dose, the constituent, and the age of the flies used.

The 50 per cent toxicity curves show the knock-down portion as a period during which the doses decrease rapidly to a minimum and the recovery portion as the period during which the insects recovering from the effects of knock-down cause an increase in the required dose.

It was found that pyrethrin II and cinerin II have higher initial knock-down powers and allow faster recoveries than pyrethrin I and cinerin I, but the relative toxicities of pyrethrin II and cinerin II decrease, while those of pyrethrin I and cinerin I increase with time, so that three hours after treatment the relative toxicities of the four constituents are pyrethrum extract 1.0, pyrethrin I 1.10, pyrethrin II 1.50, cinerin I 0.66 and cinerin II 0.51. The synergistic factors also vary with time, being small during the first 15 minutes after treatment, increasing rapidly during the following 90 minutes or so and then rising very slowly.

Features of early analysis

A preliminary analysis of the relative toxicities and synergistic factors of the four constituents and the extract at selected time intervals shows the following features:

1. The synergist increases the number of insects affected, prolongs the period of maximum effect and slows down and reduces recovery to a very considerable extent.
2. The cynerins are synergised more effectively than the pyrethrins, and the chrysanthemic esters better than the pyrethric esters.
3. The limited effect of the synergist on the knock-down activity of the constituents, and its large effect on the recovery of the insects from these poisons, which take place during the first few hours after treatment.

Other studies made by the institute during the year were concerned with the possibilities of a nicotine extraction plant (now discontinued) and of methods of sampling for pyrethrum (made necessary through discrepancies in results obtained by buyers and sellers).

American studies to control stable fly

THE STABLE FLY, *Stomoxys calcitrans* (L.), is one of the most damaging insect pests of livestock in many parts of the world. It cannot be controlled satisfactorily with insecticides and techniques commonly used to control horn flies and house flies. The reasons for this are that (1) stable flies spend very little time on animals, (2) they feed primarily on the legs of animals where insecticides are subject to being rubbed off or covered with mud, and (3) the adults seldom enter buildings to rest.

The Nebraska Agricultural Experiment Station and the U.S. Department of Agriculture are co-operating in studying the biology and control of this pest. Observations have shown that the flies prefer to lay their eggs outside of buildings on wet, decaying hay or straw. Livestock owners frequently overlook these areas, as they are are often outside of cattle pens.

Research has shown that stable flies wander from the place where they develop. Marked flies were captured and identified more than three miles from the spot where they were released. This suggests that stable fly control should be a widespread project involving destruction of breeding sites.

Encouraging nature

Studies have indicated that parasites which destroy flies by depositing their eggs in the fly pupae may be an important way that nature reduces the number of flies, and work is now going on to see if this process can be encouraged further.

New insecticides which are relatively non-hazardous to human beings and animals are being investigated as control measures.

New 'broad spectrum' worming drug

MINTIC, a drug which is said to enable farmers themselves to treat their cattle and sheep with an oral drench against the whole range of intestinal roundworms causing parasitic gastro-enteritis, is announced by Imperial Chemical Industries Ltd. It has been developed by I.C.I.'s pharmaceuticals division as a result of their successful introduction in February this year of Promintic, an entirely new worming drug for administration as an injection by the veterinary profession.

Methyridine, the basic chemical in both products, represents a marked advance on other treatments, including phenothiazine. Its most important feature, say I.C.I., is that it attacks stomach and intestinal worms at all stages in the life cycle.

Another important feature of Mintic is its broad spectrum of activity. Unlike phenothiazine, which is a valuable drug in the treatment of certain stages of worms, Mintic has a wide range of activity and will thus make up for some of the deficiencies of phenothiazine. Mintic is not, however, active against tape worms or liver fluke.

Administered as a drench, Mintic is said to be absolutely safe at the recommended dose. It has no undesirable side effects and no traces are left in the body after twenty-four hours. It does not stain the fleece nor discolour milk.

The cost of Mintic to the farmer in the U.K. will depend on the age and weight of the animal to be treated. For example, the cost of dosing a full-grown ewe of the heavier breeds will be 2s., and for the smallest lamb 4d. For a yearling calf weighing about 3 cwt. the cost would be 6s.

Demonstrating bird repellent in N. America

MR. J. E. FYNN, special products and export manager of Rentokil Products Ltd., of Leatherhead, Surrey, was visiting North America last month with the purpose of demonstrating the applications of the company's plastic gel bird repellent, Scarecrow Strip.

His first calls were in the USA (where the product is being manufactured under license by Neil A. Maclean Co. Inc. of California). In New York, he gave demonstrations on three buildings by arrangement with J. J. Hess Exterminating Services Corp., in Philadelphia in conjunction with the Theodore Meyer Exterminating Co., and in Chicago with W. B. McCloud Co. Mr. Fynn then travelled on to Toronto and Montreal for further demonstrations. This month (October) he was due to make calls in Paris.

Successful use of Scarecrow Strip is reported by Rentokil in many parts of the world, including Copenhagen, Oslo, Johannesburg, Durban, in the Persian Gulf (on marine oil drilling structures), and in Perth, Melbourne and Sydney (in Australia the strip is called Bird Off).

A disinfestation operator applying Scarecrow Strip to a ledge using a caulking gun.

Concentrated wormkiller

A new concentrated wormkiller has been added to the Supplex range of horticultural products, sold by F. W. Berk & Co. Ltd., of London. Supplex wormkiller contains Chlordane, which is also effective with leatherjackets, ants, chafer grubs and other pests. It is highly concentrated, one pint being sufficient to treat a tennis court, at the rate of 4,000 sq. yds. per gallon.

The wormkiller is available in both liquid and granular form. The liquid, a 75 per cent emulsifiable concentrate, can easily be applied by spray, and requires no watering in, say the manufacturers. The granular form can be distributed by means of a fertiliser spreader. Both forms may be applied simultaneously with other turf treatments. The wormkiller destroys worms underground, so that no sweeping up is required, and remains effective for a considerable period, irrespective of above-ground conditions.

Supplex wormkiller is at present available only in quantities appropriate to large users, but individual packs will be introduced during 1962.

Government studies of pesticide toxicity

BECAUSE OF the extra work made necessary by investigations into the toxicity of certain pesticides and their possible dangers to wild life, the Government Chemist (the British government's analytical service) has arranged for a new subdivision to undertake this field of work. In the annual report for 1960 from that laboratory, it is said that the study of this question has called for so much attention that it has been interfering with other work.

Developments during the year included further investigation of methods for the determination of traces of pesticide residues in crops and other foodstuffs; most of this work is done for the Scientific Sub-Committee of the Interdepartmental Advisory Committee on Poisonous Substances used in Agriculture and Food Storage.

In collaboration with the MAFF Infestation Control Laboratory, an examination has been made of the flesh and viscera of a number of pigeons fed with known amounts of dieldrin, while samples of the flesh and organs of captive foxes have been made available by the Royal Veterinary College. The report says that observations show that the post mortem level of dieldrin in the flesh when death resulted from dieldrin poisoning varies somewhat; the critical ranges appear to be between 10 and 15 p.p.m. dieldrin in the flesh. The work on pigeons and foxes is still in progress.

The report describes methods used for sample and testing for residues derived from fluoroacetamide treatment, and from organomercury fungicides, and organophosphorus insecticides.

"Biological methods of control are best"

Measures for minimising the effect of toxic pesticides on wildlife are suggested by Dr. John L. George, of the Patuxent Wildlife Research Center of the U.S. Fish and Wildlife Service, in an article recently in the British weekly, *New Scientist*.

He says that there are many undesirable aspects of pesticide use, and people who question the automatic use of chemical treatment should not be regarded as obstructionists. He calls for more study devoted to biological methods of control, for "control of insects by other insects and by disease is probably the most important factor in regulation of insect numbers, despite all the use of pesticides". Probably all insects could be controlled by biological means, at least to a degree, says Dr. George. Judicious chemical control as a supplement to biological control could probably control many insect pests more cheaply than could chemicals alone.

He gives six precautions which should be taken to minimise wildlife damage:

1. Use chemical treatment only when entomological research has proved it to be necessary.
2. Before pesticides are used, the effects on different kinds of animals and on animals living in different habitats should be known and carefully considered.
3. Only minimum quantities of chemicals necessary to achieve adequate control of pests should be applied.
4. Pesticides should not be applied to areas that are any larger than is necessary and the chemicals that are used should be the ones whose effects are no more long-lasting than is necessary.
5. Whenever possible, chemicals should be applied at the seasons of the year when wildlife damage will be least.
6. Serious effort should be made to be sure that pesticides are applied at no more than the intended rates and that no areas receive double doses. In large-scale treatments, it is very difficult to avoid areas of overlap.

Dr. George suggests that the "greatest promise of all" is regarded by many research entomologists as being the development of strains of plants and animals which are resistant to insects and disease.

Australian wheat export plans

Plans which could lead to the inspection of all Australian wheat for export, to ensure freedom from insect pests, have been endorsed by the Australian Agricultural Council.

The Minister for Primary Industry, Mr. C. F. Adermann, said that inspection of wheat for export had so far been successful and there was a greater demand from overseas countries for phytosanitary certificates. As a result, the Australian Wheat Board has requested that all wheat for export be covered by export regulations.

Mr. Adermann said it was also necessary to inspect the holds of ships for pests. However, the Council of Marine Underwriters had advised that its surveyors were unable to undertake certification of ships as being free from infestation, and it had been suggested that regulations be introduced to give the Federal Government power to inspect cargo vessels.

State authorities recognised the need for continued inspections to ensure that standards of export wheat were such as would meet the requirements of overseas markets. However, the cost of this work was a burden on limited State resources, and the authorities concerned considered that some financial reimbursement to the States would be required to meet the cost of staff needed to inspect wheat, Mr. Adermann said.

Rabbit control

CONTROL of rabbits is costing more than £1 million a year, says the annual report of the New Zealand department of agriculture.

For the year ended March 31st, 1961, the government contributed £530,499 in subsidies on rabbit board rates, and £115,000 in grants. There are now 208 rabbit boards controlling infestation on 39,500,000 acres in New Zealand.

Cuprinol price changes

CUPRINOL LTD. have announced changes in the prices of some of their lines following increases in taxation on solvent and fuel oils.

Retail prices in the wood preservative grades have been increased by 6d. per gallon, and by 3d. on the ½ gallon and below. Trade prices have been raised by 6d. per gallon on the 5-gallon container sizes and 3d. per gallon on the 40-gallon drums.

The increases in the woodworm Killer grades, standard and special duty, are the same as for the wood preservatives in the 5-gallon containers and 40-gallon drums. In the retail selling prices, there is an increase of 9d. per gallon and 3d. per ½ gallon and some of the smaller sizes for standard, and 1/- per gallon for special duty.

Prices for Heavy Duty Brown, Cuprinol for brickwork, Cuprinol silicone wax polish, Bilgex and the fungicidal paints remain unchanged.

Sheep scab regulations

BECAUSE SHEEP scab (which was once a serious problem in England and Wales) has now virtually disappeared in the U.K. except for occasional animals found at ports of entry, the Ministry of Agriculture, Fisheries and Food is asking local authorities to consider revoking their existing local regulations about sheep dipping. The Sheep Scab Order 1958 enables the MAFF to detain affected sheep and their contacts, to place restrictions on the movement of all sheep within a given area, and to require them to be dipped. Local authorities are also empowered to make similar regulations, and a large number of county councils and boroughs are evidently in favour of keeping them in force.

Microsol licence

LICENCE TO manufacture and distribute Microsol pest control equipment on a world basis, with the exception of North America and Italy, has been obtained by the UK company, Citenco Ltd., of Manor Way, Boreham Wood, Herts.

To facilitate the handling of this activity, the company has announced that it has set up a new Microsol division, which is to be headed by Mr. K. R. Hill.

Initially, says the company, it will be making the model 202, and next year it is expected that the model 303 will be included in the production programme.

New company

MASSEY-FERGUSON of Coventry, England, and the Butler Manufacturing Co. of Kansas City, U.S.A., have announced that a new company, Massey-Ferguson-Butler Ltd., has been formed with an equal shareholding, to market the well-known range of Butler designed grain handling, drying and storage facilities, bulk feed bins, and farm buildings in the United Kingdom and overseas.

The Butler Co. has been established for sixty years in North America in the farmstead market.

Some components will be imported from Butlers' Canadian manufacturing plant as an introductory measure but Massey-Ferguson is formulating plans to manufacture Butler equipment in the United Kingdom for the British domestic market and also for export.

Pyrethrum discounts

NEW DISCOUNTS on the prices of Pyrethrum extracts have been announced by the Pyrethrum Board of Kenya. A label discount amounting to 5s. 6d. per pound of Extract 25% is available to manufacturers including pyrethrum as an activator in formulations based on synthetic residual toxicants.

The "symbol discount", which amounts to 9s. per pound of Extract 25% is available to manufacturers whose formulations contain only pyrethrum and which are of sufficiently high quality to conform with minimum standards laid down by the Pyrethrum Board and where the manufacturer includes in his label the new symbol for African pyrethrum.

About £60,000 will be spent this year advertising the symbol to the public.

Full details of the board's scheme may be obtained on application from Biddle, Sawyer & Co. Ltd., London, the board's sole distributors in this country.

Orders are reported to be in hand for the sale of Kenya pyrethrum worth £2 million during the next twelve months to ten European countries. In addition, further contracts worth more than £500,000have already been arranged for the supply of extract to a number of other countries, including Australia, South Africa, South America and the Far East.

OFFICIAL APPOINTMENTS

ENTOMOLOGIST

REQUIRED by the West African Institute for Trypanosomiasis Research to carry out Laboratory and field investigations on tsetse flies, at Kaduna Northern Nigeria. Appointment on contract for two tours of 12-24 months in the first instance.

Salary according to qualifications and experience in scale £1,248 a year rising to £2,820 a year. Gratuity at rate of £150 a year payable at end of a tour or on final completion of services. Free passages for officer and wife. Assistance towards childrens' passages and/or grant up to £150 a year if educated in U.K. Liberal leave on full salary. Quarters provided at moderate rental.

Candidates must possess a good honours degree in Biology, with Entomology as a special subject, and have had not less than two years post-graduate research experience in Entomology. Women candidates must be single.

Apply to CROWN AGENTS, 4 Millbank, London, S.W.1., for application form and further particulars, stating age, name, brief details of qualifications and experience and quoting reference M3B/53514/PBN.

WHO bilharzia studies

The leader of the World Health Organization Bilharziasis Advisory Team, Dr. D. B. McMullen recently returned to Geneva, after spending a fortnight in Tanganyika visiting places where bilharzia is a major problem, or where it might be expected to become so with the development of irrigation schemes. Throughout his visit, he was accompanied by Dr. P. Jordan, Acting Director of the East African Institute for Medical Research.

The experts were very much interested in the work being carried out at the East African Institute for Medical Research, and had discussions in Mwanza and Arusha with representatives of the Water Development and other departments.

Another place of interest was the Tropical Pesticides Research Institute where the work being done on molluscicides was discussed.

The doctors spent two days at the Tanganyika Planting Co. at Arusha Chini and made certain recommendations to the management. They also flew to Mbeya to see the Mbarali Irrigation Scheme. Although the channels there are not yet infested with snails, it is felt that this is only a question of time and in view of the new schemes for irrigation in the area the very early consideration of bilharzia problems is essential.

World study of wood

The FAO of the United Nations has announced that its forestry and forest products division has decided to form a continuing working party on wood preservation. The British Wood Preserving Association is among those bodies which have been invited to participate in its activities.

A preliminary programme of work has been drafted, as follows:

1. Nomenclature, classification and definitions. Initial objectives are to prepare recommendations on nomenclature and definitions relating to fungi, insects and other destructive organisms, and definitions of terms used on wood preservation for both products and processes.

2. Correlation of experience from stake and service with laboratory tests.

3. Compatibility of glueing and wood preservation materials.

4. Laboratory tests on the influence of wood preservatives on metal corrosion.

5. Stake tests. (The purpose of activities under this heading is to investigate how far recommendations on stake tests are required).

6. Unification of panel tests for evaluating natural durability and efficiency of preservatives against marine borers and fungi.

7. Preparation of leaflet on wood preservation in housing. This leaflet is intended for distribution in countries of Africa, the Asia-Pacific region and in South America.

8. Specification manual on the treatment of timber for housing.

New rooting powder

A double action rooting powder which gives increased rate of "strike" and protection from soil-born diseases is the latest addition to Boots the Chemists range of gardening aids.

Boots Hormone Rooting Powder is a formulation containing the rooting hormones a-Naphthaleneacetic acid and B-Inaolybutyric acid, together with the fungicide T.M.T.D. It is said to encourage cuttings to root quickly and vigorously, and also to give protection from stem rots and other soil-born diseases, while rooting is taking place.

Research overseas

Britain spent nearly £26 million—another record year—on development and welfare schemes to benefit her overseas territories in the last financial year. This is revealed in the annual "Return of Schemes" just published in London.

Of this figure, it is shown that £223,784 was spent for work on tsetse and trypanosomiasis research in various territories.

Do's and don'ts with sprays

The Ministry of Agriculture in Britain has published two leaflets about the correct way to use chemical sprays. The leaflets have been prepared by the Ministry in consultation with the organisations of the chemical manufacturers, merchants, contractors, farmers and agricultural workers.

The leaflet, *Do's and Don'ts for Users of Sprays*, lists the good and bad practices in spraying and is accompanied by a wall card for the special attention of agricultural workers. The *Code of Practice for Ground Spraying* gives more detailed advice on the use and maintenance of spraying machines, correct field practice and ways to overcome the hazards associated with the use of chemicals.

Balloons and birds

The suggestion for using balloon barrages to protect orchard crops from birds (in a similar manner to those used for protecting British cities from dive-bombing attacks in the last World War) has been made at the recent union conference of the Australian Apple and Pear Growers' Association in Adelaide.

One delegate said that the birds were destroying up to one-third of crops in the state of Victoria, and that starlings were the worst offenders. He said that there were several growers he knew who had used balloons to keep birds away.

New appointments

Dr. F. C. Lloyd, B.Sc., F.R.I.C., has joined F. W. Berk & Co. Ltd., the London chemical manufacturers, in the position of development director, although not a member of the main Board. Dr. Lloyd was formerly research manager of B.X. Plastics Ltd.

Mr. Norman J. Travis and Mr. James D. Tennant, directors of Borax (Holdings) Ltd., have been appointed to the board of Hardman & Holden Ltd.

Mr. C. A. Lister has been appointed Commercial Manager of Shell Chemical Company's Agricultural Division. He joined the company from Shell International Chemical Company where he was Area Co-Ordinator for Australasia and South-East Asia.

NEW PUBLICATIONS

The Economic Plants Diseases of Scotland. *By C. E. Foister. Published by Dept. of Agriculture and Fisheries for Scotland. (H.M. Stationery Office. Price 10s. 9d.).*

The diseases which affect many Scottish plants are described in a new bulletin which has been written by Dr. Foister, director of the agricultural scientific services of the the department, and which is No. 1 in a new series of technical bulletins.

It was produced specially as a work of reference for advisory officers in the U.K., and for the plant protection officers of foreign countries following the 1951 International Plant Protection Convention which urged all countries to report on the diseases of their economic plants.

Part I of the Bulletin deals with the cropping in Scotland, and the distribution, economic importance and yearly occurrence from 1924 to 1957 of all diseases caused by bacteria, fungi, viruses, non-parasitic agents, deficiencies and toxic chemicals etc. The crops are dealt with under the headings of potato, cereals, roots, fruits, vegetables, pasture, forage and pulse, ornamentals, shrubs and forest trees. Part II is a "check list" of the diseases arranged under the plant Latin names. A feature is made of describing symptoms where there is no well recognised common name for the disease. Part III is an index to all parasites, viruses and non-parasitic causes and to the English common names of the host plants. There is an extensive list of literature references and an appendix giving details of the Department of Agriculture and Fisheries' certification schemes in 1959 for potato, oats, barley, shallots, blackcurrants, raspberries and strawberries. Summarised meterological records are provided for 1942-57 for comparison with outbreaks of major diseases. It is hoped that the bulletin will also be of use to plant pathologists in general, to research institutes, university and college teaching establishments and at least to the leaders in the agricultural and horticultural producing and trading communities.

Market for Pesticides in the Central African Federation, Ghana, Kenya, Nigeria, Tanganyika and Uganda. *Published by the Colonial Pesticides Research Committee, Tropical Products Institute. 56-62 Grays Inn Road, London, WC1. Price 25s. net).*

The C.P.R.C. has been taking an increasing interest in the economics of pest control, and has now just issued a report on the market for pesticide products in these parts of Africa. Largely by means of tables, the report sets out to assess (for example) the potential market for pesticides on six main crops, i.e. cacao, coffee, cotton, groundnuts, tobacco and cereals, of which it discusses the main pests, the total area of the crop, the pesticide that could be used for treatment, and a calculation of the average rate of usage of the active ingredients. Figures for the "maximum economic potential of the market" for each product, that is, the greatest quantity that could be used were the farmers to use pesticides to their own best advantage, are reached by considering each crop and each pest in each area and the area affected sufficiently by a pest in an average year enough to warrant treatment, and indicating how farmers can use more pesticide than is economically justified.

Figures relating to pesticide imports and their estimated future potential are included.

Glamorgan Forests. *Forestry Commission Guide, edited by H. L. Edlin. (H.M. Stationery Office, London. Price 5s. net).*

This charming little guide book is the story of eight Welsh forests, which represent an important industry in Glamorgan where the Forestry Commission started planting just forty years ago. Much of it is concerned with its fascinating history and antiquities, its literature, but principally it is devoted to the long-term work of planting new forests and maintaining existing ones in the region.

TECHNICAL LITERATURE AND PUBLICATIONS

Albright & Wilson Ltd. (1 Knightsbridge Green, London, SW1). The group has produced a 70-page book showing the diverse fields of operation of its member companies. The development of each of these companies is traced and, by means of many illustrations in full colour, their products and their end-uses are are illustrated. Agricultural chemicals used in the production of insecticides are among these.

Allied Chemical International (40 Rector St., New York 6, USA). A new technical brochure evaluating seven types of polyethylene glycol has been made available. The brochure outlines specific applications in many consumer fields; in agricultural products, fatty acid esters are used to manufacture insecticidal sprays, and are also intermediates for weed killers and are carriers for other agricultural chemicals.

Pan Britannica Industries Ltd. (Waltham Abbey, Essex). Two leaflets have just been issued by this company about their products. One, which deals with their Malathexo 60, has been completely revised and contains additional recommendations for top fruit and mushrooms. There is also a low volume recommendation for glasshouse crops. Additions have been made to the list of plants which should not be sprayed with this product. The second leaflet is devoted to Combinex, and includes low volume recommendations and advice for the control of leaf miner.

Murphy Chemical Co. (Wheathampstead, St. Albans, Herts.) New leaflet just issued by the company is concerned with the control of red spider on apples and pears. It gives details of control measures and includes a note on control of fruitlet mining tortrix. Other literature just available from this company deal with the control of codling and tortrix moths, and of cabbage aphid.